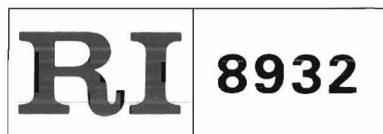


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Spectral Characteristics of Grinding Sparks Used for Identification of Scrap Metals

By W. D. Riley, B. W. Dunning, Jr., and D. M. Soboroff



UNITED STATES DEPARTMENT OF THE INTERIOR



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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

| | | | |
|--------|-----------------|--------|----------------|
| ft/min | foot per minute | ms | millisecond |
| in | inch | nm | nanometer |
| μm | micrometer | s | second |
| μs | microsecond | wt pct | weight percent |
| mm | millimeter | | |

SPECTRAL CHARACTERISTICS OF GRINDING SPARKS USED FOR IDENTIFICATION OF SCRAP METALS

By W. D. Riley,¹ B. W. Dunning, Jr.,² and D. M. Soboroff³

ABSTRACT

The Bureau of Mines is conducting research on methods for the rapid identification and sorting of mixed scrap metals and alloys so that domestic scrap resources can be fully utilized. One method, spark testing, is the examination of the pattern of sparks that results when a metal or alloy is ground on an abrasive wheel. This technique requires experienced sorters. Bureau researchers have shown that spectrophotometric methods can be used in lieu of the highly skilled sorter. The spectra of a mild steel, an HSLA steel, several stainless steels, and four nickel-base alloys have been measured. Differences in the spectra that are sufficient for identification are apparent.

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INTRODUCTION

Conservation of strategic metals and minimizing imports through improved methods of recycling scrap materials are among the objectives of the Bureau of Mines Minerals and Materials Recycling program. Since the first step in conventional recycling technology for metals and alloys is to segregate the mixed scrap into groups of like materials, an important aspect of this work has been the evaluation and introduction of new technologies to identify and sort scrap metals (1, 12, 15).⁴ Conventional techniques such as chemical spot testing, determination of magnetic properties, and spark testing still play a major role in the sorting of scrap metals in many scrap yards.

Spark testing requires observation of the pattern of sparks that results when a metal or metal alloy is ground on an abrasive wheel. Sparks occur because most metals and alloys, in the finely divided state, oxidize rapidly when heated to incandescence. When metals or alloys are ground using a high-speed grinding wheel, the fine particles torn loose are heated by the frictional forces of the wheel and by overcoming rupture resistance. As the metal or alloy reaches incandescent temperature, it burns. The resulting pattern of sparks can be used to identify many metals and alloys by examining the spark pattern for shape of spark stream, color, and various characteristics that are imparted by alloying constituents. There have been attempts to standardize this technique by specifying the type of abrasive wheel, its speed, and the type of lighting that should be employed in the working area (2, 7, 11); however, the identification is still very dependent on the skill and experience of the tester.

Figure 1 shows some of the characteristics that are found in a spark stream whose flight path is from right to left (12). For example, in iron-base alloys;

the frequency of bursts is related to the carbon content, with the intensity and size of bursts increasing with increasing carbon content. Noncarbide-forming elements like manganese, nickel, and cobalt suppress spark bursts. Carbide-forming elements like molybdenum, chromium, and tungsten change the color of spark patterns as well as suppress spark burst. Cobalt- and nickel-base superalloys and titanium alloys also have characteristic spark streams. Descriptions of patterns have been published for various steels, superalloys (2-4, 16-17, 19), and nickel (3). Specific articles on the sorting of carbon and alloy steels have also been published (2, 8, 18). In many of these articles, different descriptions are offered for the same metal. The spark pattern of 316 stainless steel has been described as (1) short reddish spark with few spear points (16-17); (2) orange to straw colored with no spear points (4, 9); and (3) orange to straw colored with spear points (20).

Optical emission spectroscopy has been used to identify scrap materials (1, 12, 15). There also has been a study (5) that used optical emission spectroscopy

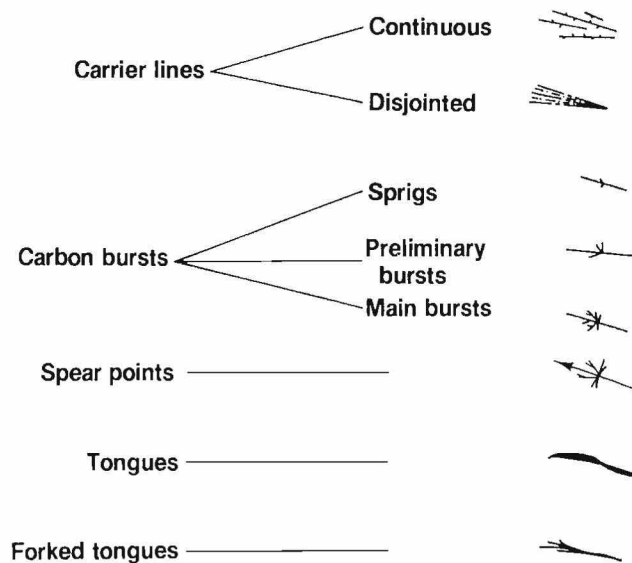


FIGURE 1. - Types of spark patterns from metals and alloys held in contact with moving abrasive wheel.

⁴Numbers in parentheses refer to items in the list of references at the end of this report.

to examine the ignition by sparks of explosive atmospheres. However, identification of alloys from the optical spectra of a spark pattern has not been previously reported. Experiments were conducted to see if spectroscopic techniques could

be used to quantify or improve this technique as a means of identifying metals and alloys (14). This report describes progress to date using several types of optical instruments.

EXPERIMENTAL EQUIPMENT AND PROCEDURE

SPECTRAL RADIANCE MEASUREMENTS

The spectra of the sparks were recorded using photodiode array optical spectroscopy, rapid-scanning optical spectrometry, and transmission filters and detectors. The detectors used in each system were most sensitive to the visible range of light wavelengths based on the assumption that color, hue (degree of chromaticity), and brightness were the most important factors in sample identification. The ultraviolet and infrared regions are not included in this study.

The photodiode array optical spectrometer has been described in detail elsewhere (5), a general description is as follows: The photodiode array consisted of 1,024 photodiodes on a section of 25-mm silicon chip. The detector was attached to a spectrometer. In this configuration, a 300-nm window can be examined with the photodiode array. For maximum sensitivity, a 250- μ m slit width was used. Exposure time was fixed at 0.5

s. The detector and spectrometer system is controlled by a signal analyzer with spectrometer module. Data reduction and storage is performed with a microcomputer.

The rapid scanning spectrometer system (fig. 2) used a silicon detector for spectral detection and a spectrometer. The wavelength scan rate was fixed at 10 nm/ms. Data were displayed on an oscilloscope equipped with dual tracing unit, one trace displayed the spectra while the other displayed the wavelength. The spectrometer was also equipped with a wavelength marker, a scan control unit, a sample and hold unit, and recorder output. This system could be configured to either display an entire spectrum or intensity at a fixed wavelength. Data reduction was accomplished on a microcomputer by digitizing the recorder output with a digitizing tablet.

The transmission filter and silicon detector system (fig. 3) consisted of three

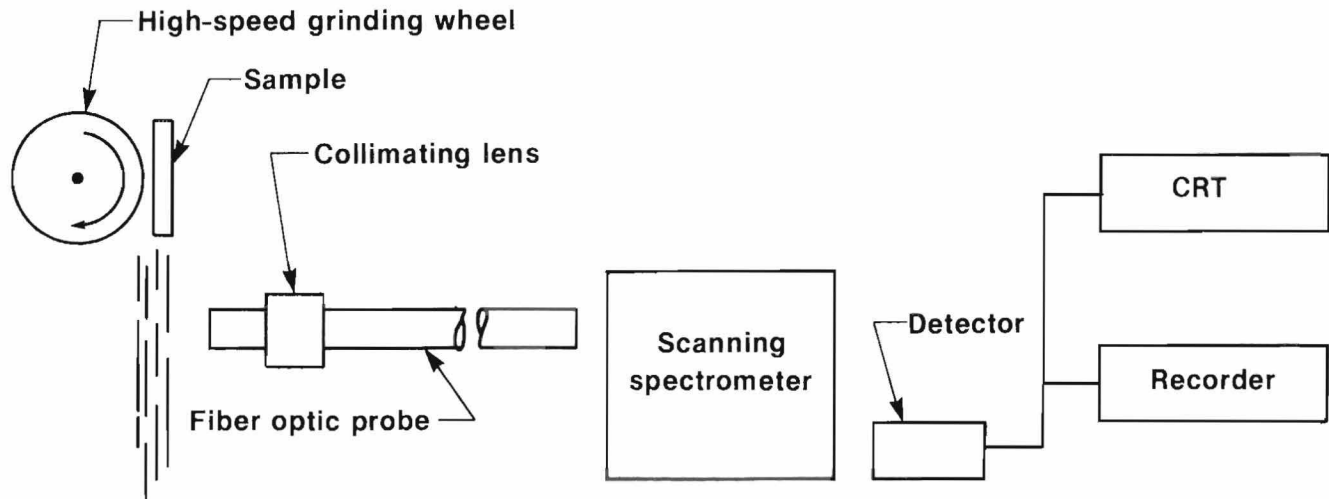


FIGURE 2. - Schematic representation of rapid-scanning spectrometer system.

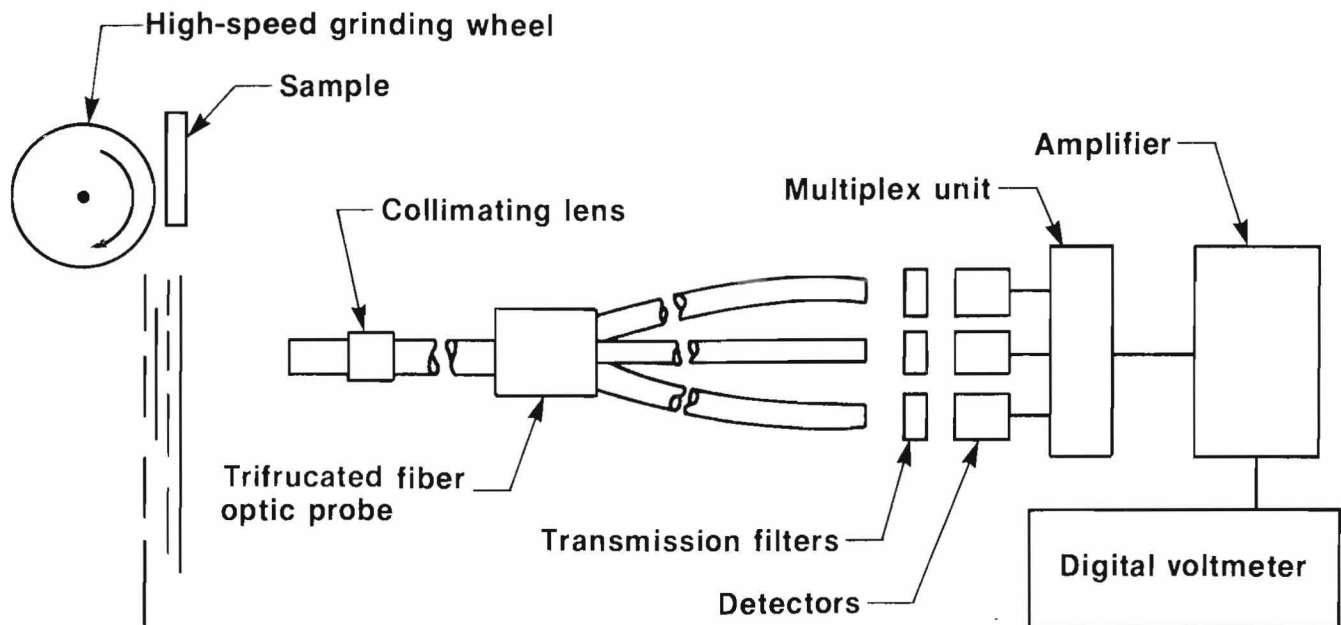


FIGURE 3. - Schematic representation of transmission filter-and-silicon detector system.

transmission filters (transmission centered at 450, 550, 650 nm), three silicon detectors, a trifurcated fiber optic cable, and a multiplexor-amplifier digital readout package. This unit displayed the intensities at the wavelengths transmitted through the filters.

Table 1 lists the degree of complexity, approximate costs, and ease of use of the various systems discussed in the paper. The most easily used system would be a rapid scanning spectrometer coupled with a computer to control all the functions, including identification of the metal.

The sparks were generated by holding a sample against a grinding wheel equipped with either an 8-in aluminum oxide wheel (medium grit) or an 8-in carborundum (al-oxite) rotating at approximately 7,500

ft/min. The wheels were dressed prior to the examination of each spectra to insure that there was no contamination from previous samples. Variations in intensity due to physical differences were minimized by holding the speed of the grinding wheel constant and the flatplate sample size to 3- by 1.5 in and by fixing sample angle of contact. The pressure of the sample (as suggested by standard spark testing convention) was adjusted to the point where the sample stopped bouncing on the wheel. Therefore, hard alloy samples required slightly more pressure than did soft alloy samples.

PHOTOGRAPHING SPARK PATTERNS

Several attempts were made to photograph the spark patterns. The best photographs were obtained by using 400

TABLE 1. - Spark-identification systems

| Equipment | Cost | Equipment | Ease of use |
|--|----------|-----------|-------------|
| Transmission filter and silicon detector.. | <\$1,000 | Simple... | Moderate. |
| Photodiode array optical spectrometer..... | 60,000 | Complex.. | Complex. |
| Rapid-scanning spectrometer..... | 2,500 | ...do.... | Moderate. |
| Rapid-scanning spectrometer w/computer.... | 7,000 | ...do.... | Simple. |

ASA color or black-and-white film in an autoexposure camera (shutter priority). Film brand made little difference. The room was darkened and the camera was set for a 0.30-s exposure so that time-averaged pictures were obtained. As figure 4, shows, much of the detail of the

spark pattern is easily seen. The flight of the sparks is from right to left.

SAMPLE PREPARATION

The 3- by 1-1/2-in metal samples listed in table 2 were prepared by degreasing in

TABLE 2. - Alloys used in study of spark-pattern characteristics

| Alloy | Major alloying elements, wt pct (nominal) | | | | | Other elements |
|-------------------|---|-----|-----|-----|-----|----------------|
| | Fe | Ni | Cr | Mo | Ti | |
| 201..... | 70 | 5 | 18 | NS | NS | Mn |
| 301..... | 73 | 7 | 17 | NS | NS | Mn |
| | 72 | 9 | 18 | NS | NS | Mn |
| 304..... | 69 | 9 | 19 | NS | NS | Mn |
| 316..... | 72 | 10 | 16 | 2.5 | NS | Mn, Si |
| 410..... | 85 | NS | 13 | NS | NS | |
| Hastelloy..... | 5 | 62 | NS | 28 | NS | |
| Incoloy 800..... | 46 | 32 | 21 | NS | NS | Mn |
| Inconel 625..... | 2.5 | 61 | 22 | 9.0 | 0.2 | Cb, Al, Mn |
| Inconel 600..... | 8.6 | 75 | 16 | NS | NS | Mn |
| 1020 CS..... | Bal. | NS | NS | NS | NS | Mn, P, S, C |
| HSLA steel..... | Bal. | .25 | .75 | .30 | NS | Mn, P, Si, Cu |
| NS Not specified. | | | | | | |

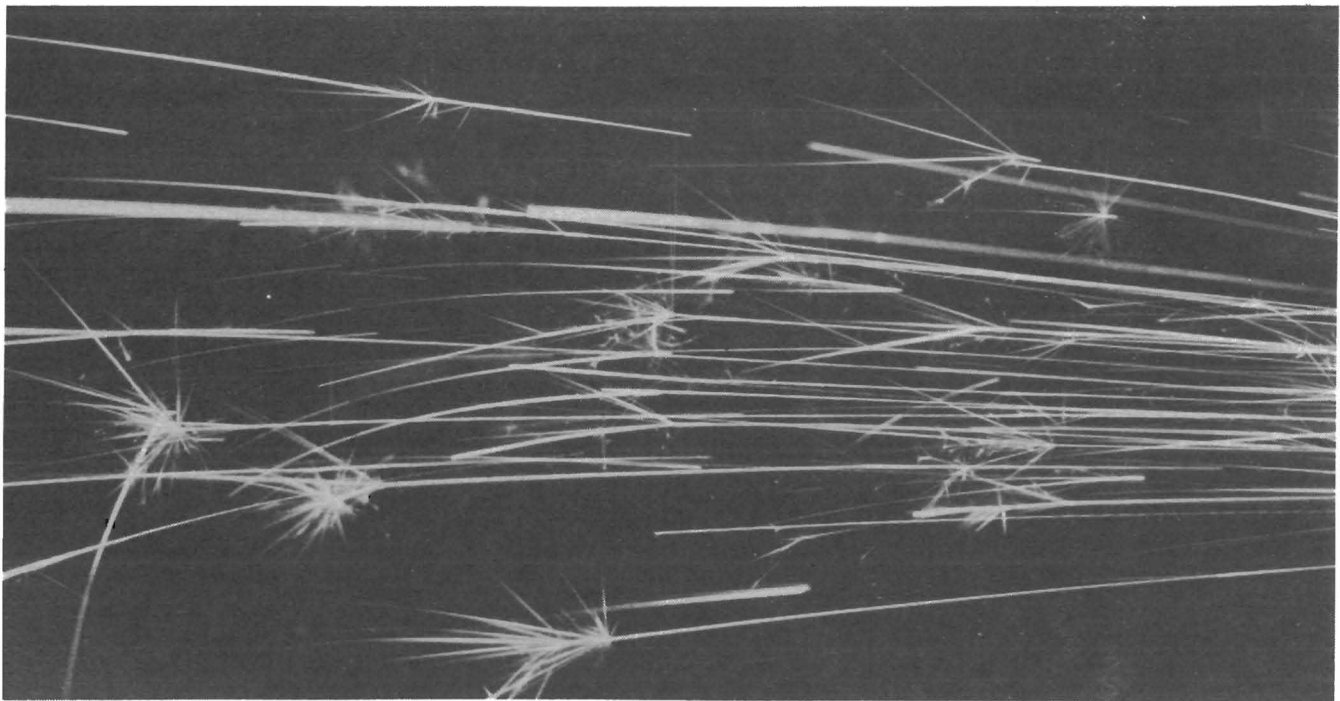


FIGURE 4. - Photograph of spark pattern for 1020 carbon steel.

acetone and light abrasion with a 120 grit silicon carbide wheel to remove any surface contamination. Typically, in the

scrap yard, the only preparation is to lightly abrade the sample prior to actual spark testing.

RESULTS AND DISCUSSION

Each of the instruments used provided a slightly different way of examining the spectra of sparks. The photodiode array optical spectrometer exposes the individual diodes to light for a predetermined amount of time. Data from the photodiode array would not show variations in the light source. When the system is used in a "one-exposure" mode, as it was in this study, the spectra displayed show the integrated intensity at each wavelength for the measurement period. The transmission filter and silicon detector system works in a similar manner. The multiplexor-amplifier is designed with an electronics package that samples the output from the silicon detector for 1 s prior to display. This time period was chosen because initial measurements made for some samples had rapidly varying voltages on the direct-current voltmeter that appeared to be "noise." The rapid-scanning spectrometer consists of an Ebert-type monochromator, which uses a continuously rotated, blazed-diffraction grating as the dispersing element. The scan rate is 10 nm/ms, and a complete scan takes 95 ms. When unalloyed carbon steels were examined using this system, it was discovered that there were very strong intensity fluctuations at specific wavelengths in the spectra. It appears that these changes are produced by the carbon bursts (star-shaped formations in figure 4), which cause an increase in radiating surface due to the particle bursting into a number of smaller fragments. Because this process is not continuous and the spectrometer is sampling a different spectral region of the spark pattern, variations in intensity with time at each wavelength are present. When the spectra of a highly alloyed, low-carbon material is examined, there are few, if any, carbon bursts, hence the curve is much smoother. The spectrum for the unalloyed carbon steel has a higher standard deviation (SD) than for 316

stainless steel (fig. 5). What appeared to be noise in the the initial transmission filter and silicon detector experiments was in reality, the fluctuations in the spectral intensity.

The spark patterns apparently contain no fine structure on the time scales used for collection. Figure 6 shows the spectrum for 316 stainless steel collected using the photodiode array spectrometer at relatively long (0.5 s) and short (0.1 s) exposure times.

Figure 7 shows the spectrum (0.5 s) for a mild steel, a stainless steel, an Inconel, and an Incoloy. A comparison of the individual spectra reveals variations in intensity versus wavelength that are attributable to composition changes. As the amount of alloying is increased from a mild steel to stainless steel to Incoloy and finally to Inconel, a decrease in the maximum intensity and a shift toward the red end of the spectrum is apparent.

Figure 8 shows the spectrum (0.5 s) for Incoloy 800, Inconel 625, Hastelloy B, and Inconel 600. Conventional literature on spark testing states that the latter three alloys have short red sparks. The differences in spectral intensity versus wavelength are evident using a spectral analyzer.

Because of the lack of spectral structure of the spectrum, a simpler instrument arrangement--the transmission filter-silicon detector system--might yield adequate information for alloy identification. The wavelengths of 450, 550, and 650 nm were selected based on the full spectral patterns obtained previously. The measurements were made by adjusting the fiber optic probe so that the light from the spark was gathered at its most intense area, approximately 2 to

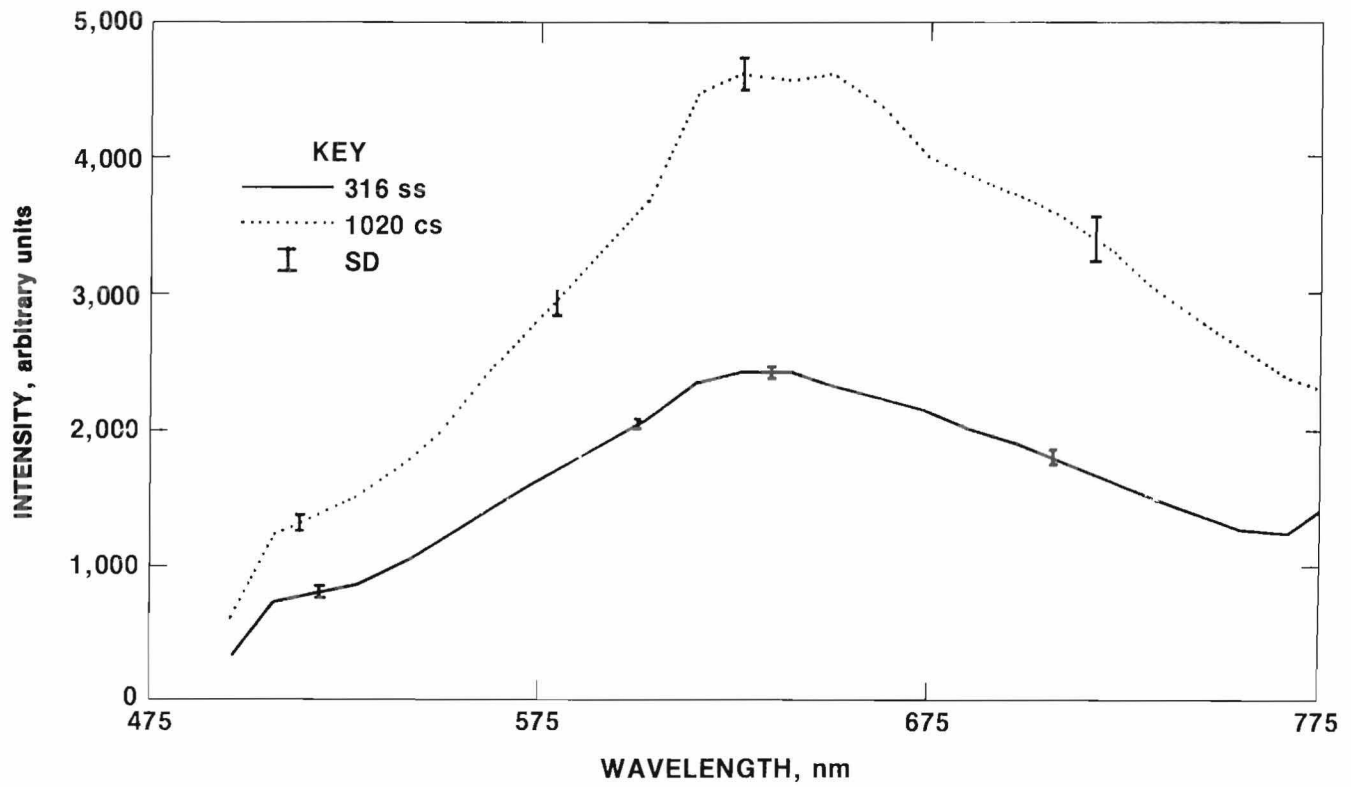


FIGURE 5. - Spark spectra of 1020 carbon steel and 316 stainless steel.

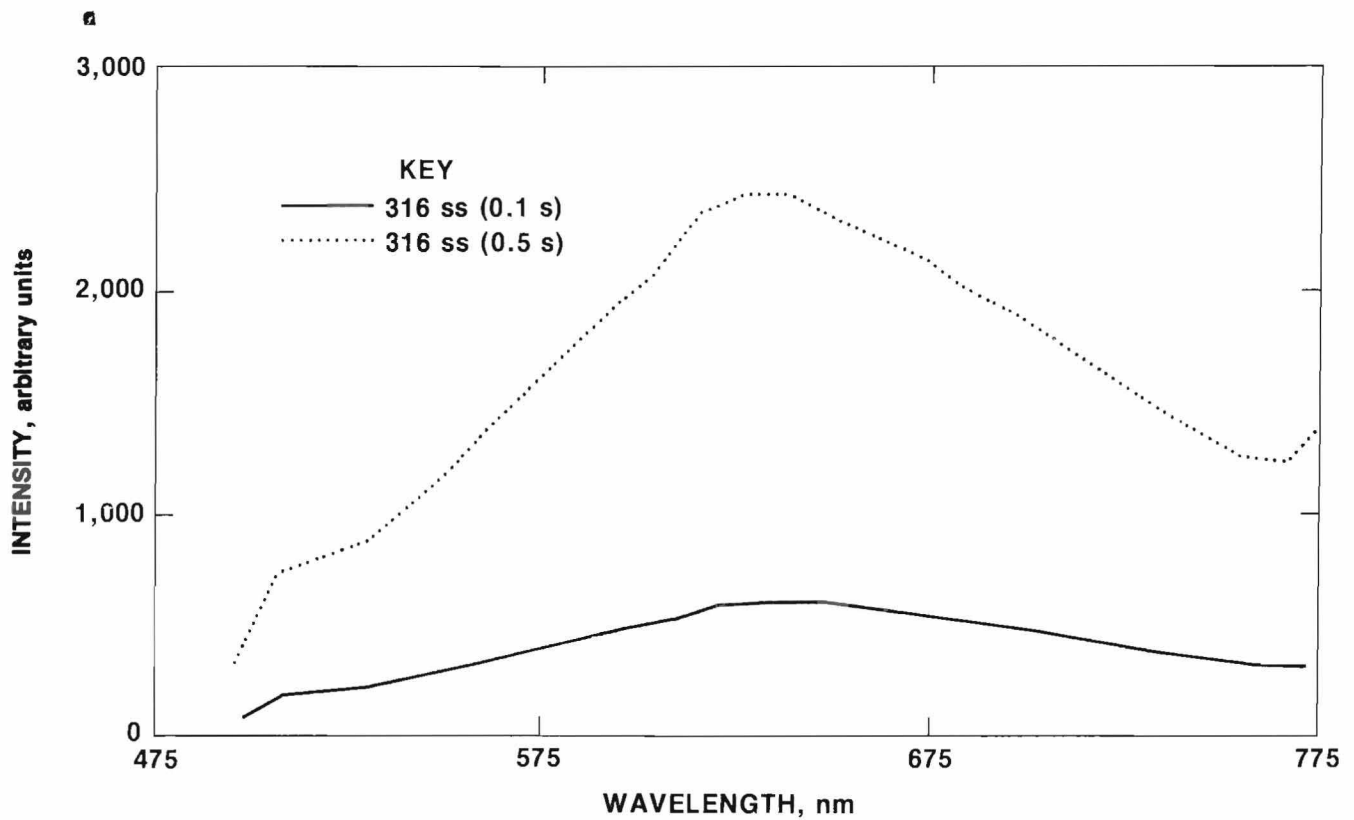


FIGURE 6. - Effect of photodiode array exposure times on spectrum of 316 stainless steel.

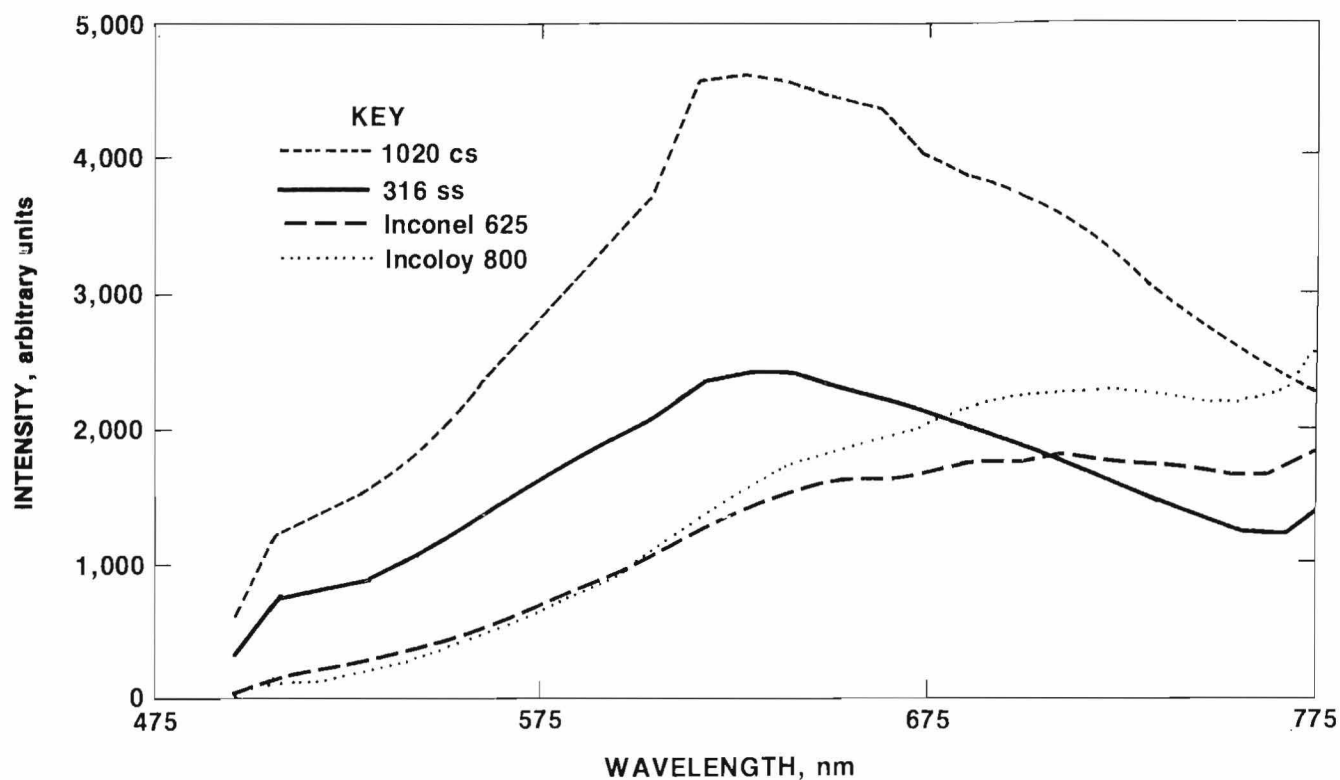


FIGURE 7. - Spark spectra for several metals showing effect of increasing nickel content using photodiode array spectrometry.

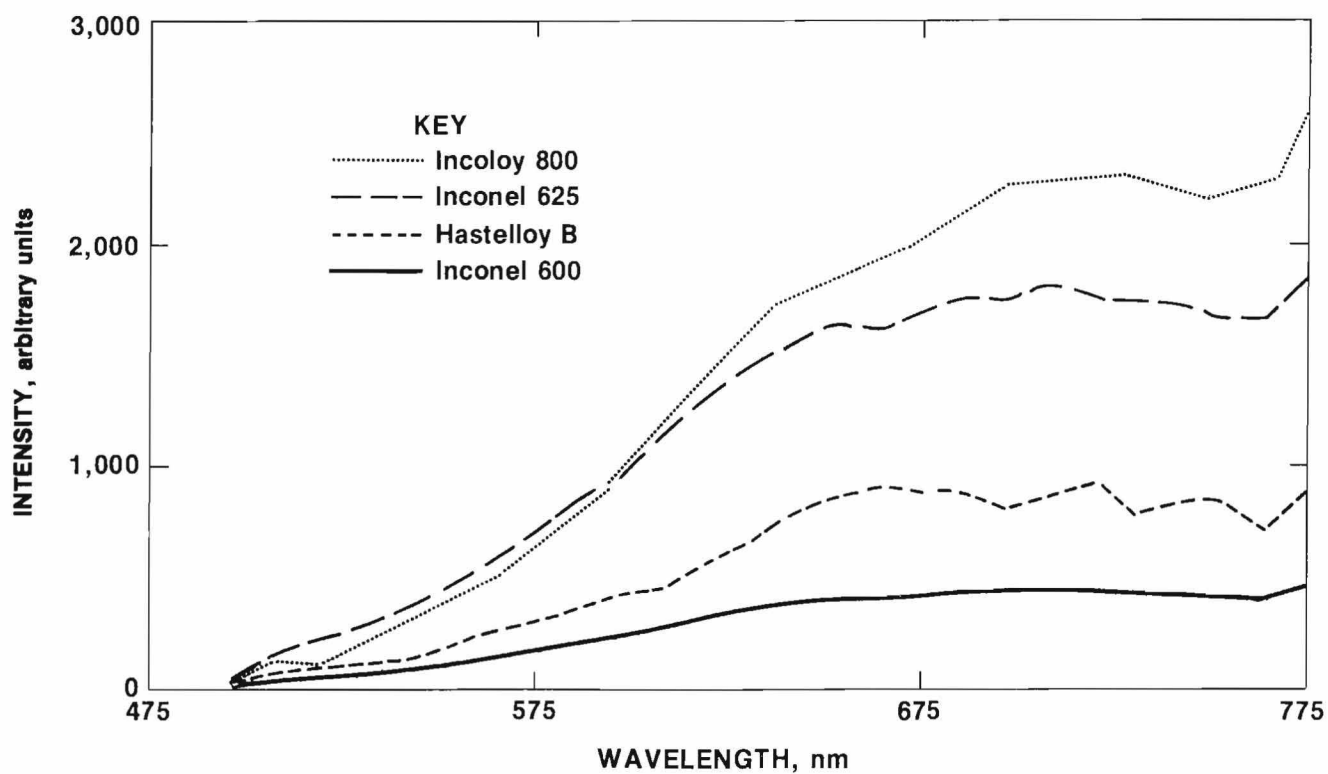


FIGURE 8. - Spark spectra for some nickel-base alloys.

5 in from the wheel. Table 3 shows the results of these experiments. The differences in the intensity values indicate that as alloying increases, so does the wavelength at which the maximum intensity occurs. Additionally, the intensity ratios show that there is enough of a spectral difference to allow the samples to be identified. Use of the ratios of the intensities at specific wavelengths corrects for variations in overall intensity observed in previous work in color sorting (11).

Spectra were also collected using the rapid-scanning spectrometer. This system (fig. 9), was more versatile than the transmission filter and silicon detector because it can show the entire spectrum

(fig. 10) or display the maximum, intensity of the spark at a particular wavelength (table 4). The results of these measurements show the same trends as the previous data. While some of the materials tested visually have different spark patterns, others such as 316 and 304

TABLE 3. - Spark intensity for various alloys, in nanometers

| Alloy | Intensity | | | Intensity ratio | |
|-----------------|-----------|-----|-----|-----------------|---------|
| | 450 | 550 | 650 | 450/650 | 550/650 |
| 1020 cs. | 0.20 | 1.4 | 1.1 | 0.18 | 1.3 |
| HSLA steel.. | .33 | 1.6 | 1.1 | .30 | 1.5 |
| 316 ss.. | .18 | .37 | .52 | .35 | .71 |
| Inconel 625.... | .04 | .15 | .38 | .10 | .38 |

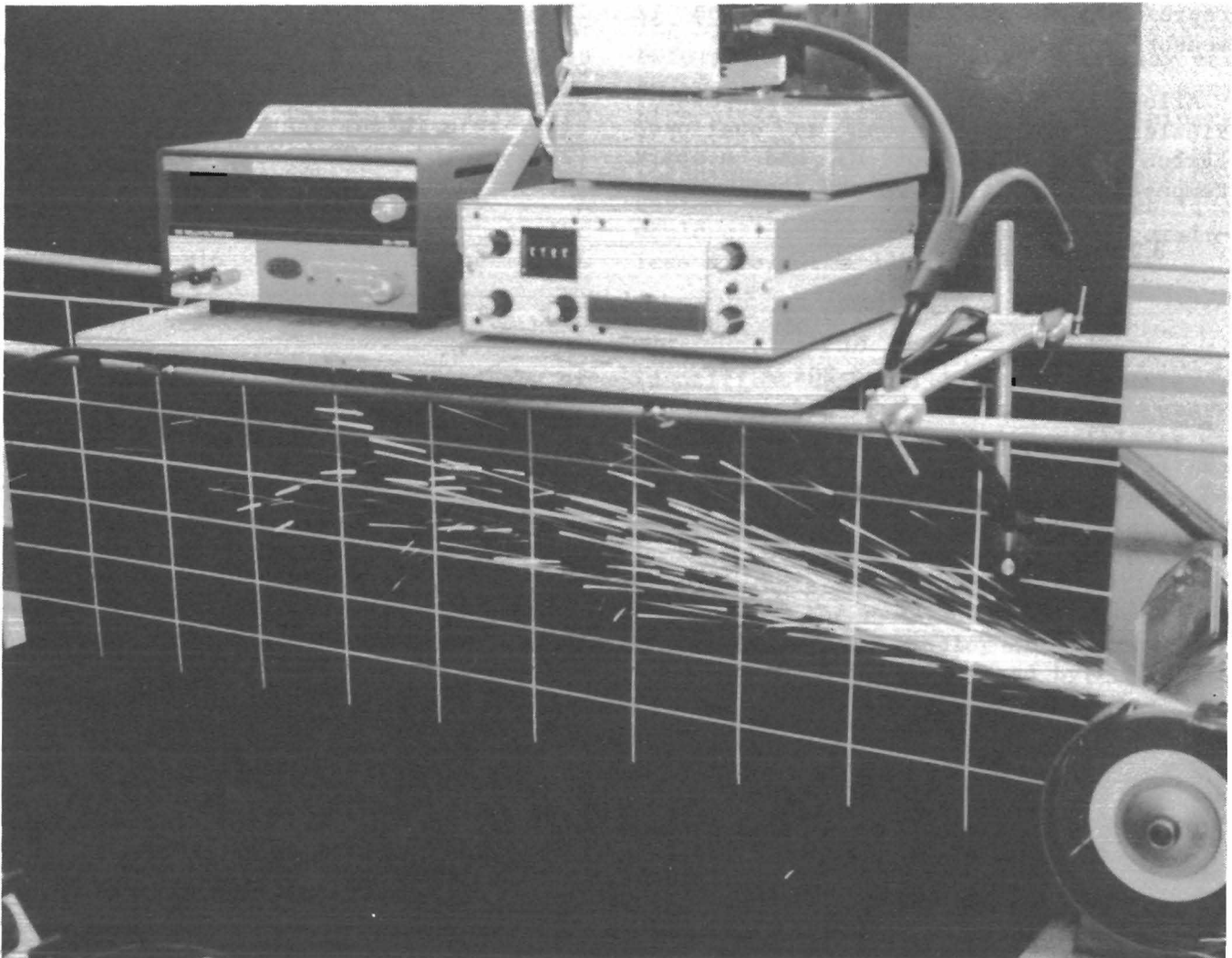


FIGURE 9. - Rapid-scanning spectrometer system.

stainless steels have very similar patterns. However, figure 10 shows, there are sufficient differences in the spectra of the alloys to allow them to be separated using this instrumental method.

TABLE 4. - Measurement of maximum intensity and wavelength

| Alloy | Wavelength, nm ¹ | Maximum intensity, arbitrary units ¹ |
|----------------|-----------------------------|---|
| 1020 cs..... | 646±2 | 97±6 |
| 410 ss..... | 651±5 | 53±2 |
| 304 ss..... | 683±3 | 39±1 |
| 316 ss..... | 672±3 | 27±1 |
| Inconel 625... | 709±2 | 20±1 |
| Incoloy 800... | 721±3 | 29±1 |

¹Numbers on the right of the symbol (±) represent the standard deviation (7 measurements).

Alloying changes the spark temperature. Riezler (13) showed that an unalloyed carbon steel (0.10 pct C) had a spark temperature of 1,640° C and that as the

carbon level increased, the temperature of the spark increased (1.00 pct C = 1,740° C). In steels containing W, Cr, and Mo, the temperature decreased as the amount of alloying increased. Riezler's measurements agree very well with the data generated in this study. Our work and that of others (8) indicate that if the alloying elements have a higher melting point than the iron matrix, then the temperature of the sparks will be lower and the color will be tinted toward red. The degree of change in color tint then depends on the amount of alloying elements. If, however, the alloying element has a lower melting point (such as Mn), the spark pattern will be both brightened and shifted toward the hotter end of the spectrum (8). Higher melting components cause larger shifts.

Several authors have suggested that the oxidation of carbon to carbon dioxide and possibly carbon monoxide plays the principal role in the spark stream of steels (2, 7-8, 10). A rapidly heated particle tends to become spherical, the surface

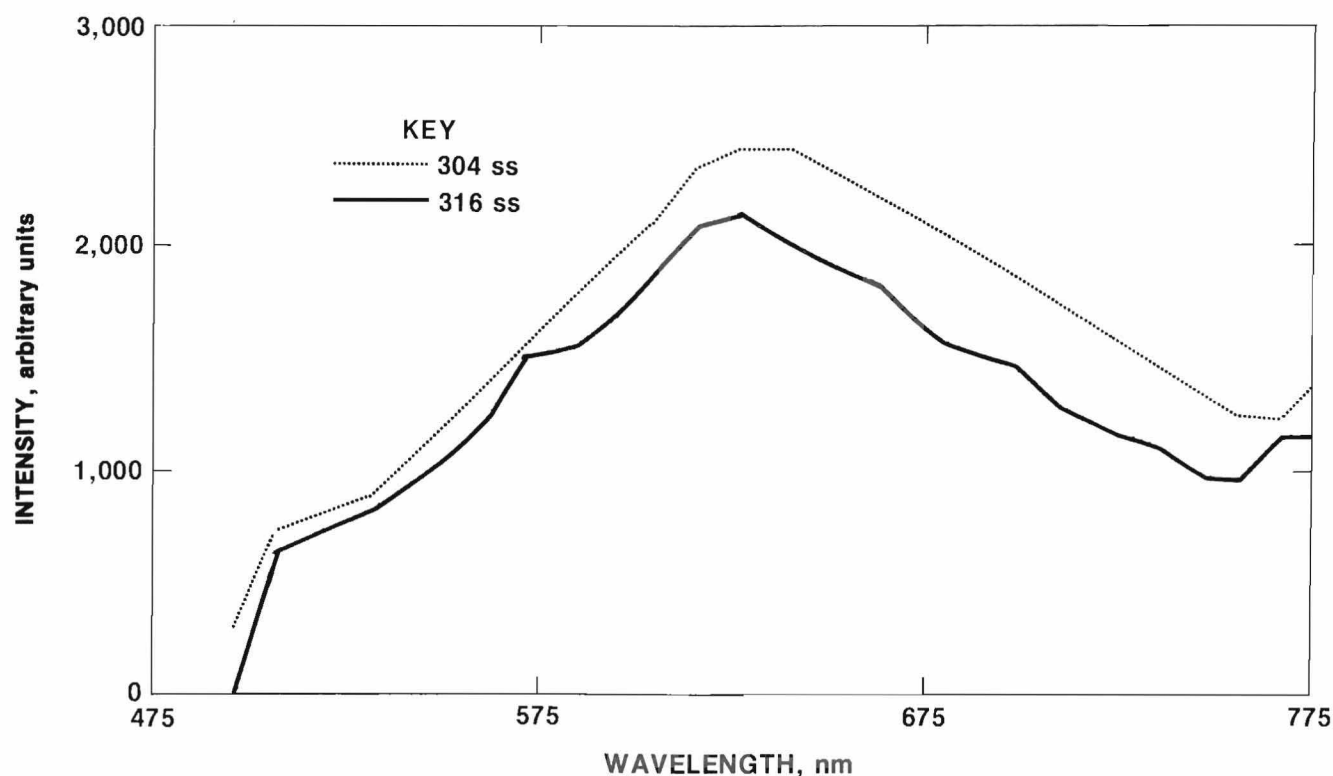


FIGURE 10. - Spark spectra of 304 and 316 stainless steel using rapid-scanning spectrometer system.

begins to oxidize to iron oxide, and the carbon is oxidized to carbon dioxide. The iron oxide easily flakes off, and iron is rather soft and plastic at the temperatures in a spark stream. Since carbon dioxide occupies a volume one order of magnitude greater than the original carbon, the gas can escape from the pellet causing the secondary bursts (2, 7-8). If this is true, it has been suggested (2) that the alloying elements might change the characteristics of the oxide film in such a way as to give the various spark characteristics.

Another explanation maybe that most of the alloying elements, except nickel, cobalt, and manganese, readily form carbides that are stable at high temperatures so that there is insufficient carbon in the matrix to form iron carbide. If we compare the pictures of 201 stainless steel (approximately 0.15 pct C) to 1020 carbon steel (approximately 0.2 pct C) in figure 11, there are very few bursts in the 201 stainless steel. The 201 stainless steel contains 16 to 18 pct Cr, which is a strong carbide former. A similar observation was made when the spectrum of 316 (0.08 pct C) and 316 L

(0.03 pct C) stainless steels were compared. The spectra are identical because the carbon was bound up as chromium and molybdenum carbides. Because the rapid rate of combustion allows no time for the carbon to go into solution in γ iron, an equilibrium does not exist. This is especially the case in the high- and low-carbon 316 stainless steels where the chromium and molybdenum carbides do not go readily into solution.

This research shows that spectroscopic examination of the spark pattern provides a means of identifying scrap metals that previously could not be identified by conventional visual spark testing techniques. For example, previous publications (4, 12) indicate that the presence of a high-nickel alloy is indicated by a short red spark and that additional testing by chemical spot testing is required before the specific nickel-base alloy can be identified. Although there are many high-nickel alloys, the preliminary indications are that use of this new technique will permit specific identification of each alloy. This technique would allow the separation of 316 stainless steel from 304 stainless steel.

CONCLUSIONS

The intensity, color, and pattern of grinding sparks are determined by the alloying elements in the sample investigated. Successful separation of alloy samples by conventional spark testing is determined by the experience of the individual performing the operation. The individual observing the sparks, develops through repetition, a memory system to recognize which metal alloys are being tested. Spectrophotometric examination of grinding sparks can be used as an

identification tool in the scrap yard, and can outperform conventional spark testing, eliminating the possibility of human error and even identifying alloys in groups that can not be separated by conventional means. The results of preliminary experiments show that even materials whose spark patterns appear similar to the eye such as 304 and 316 stainless steels, Incoloy 800, and Inconel 625 can be distinguished by this new method.

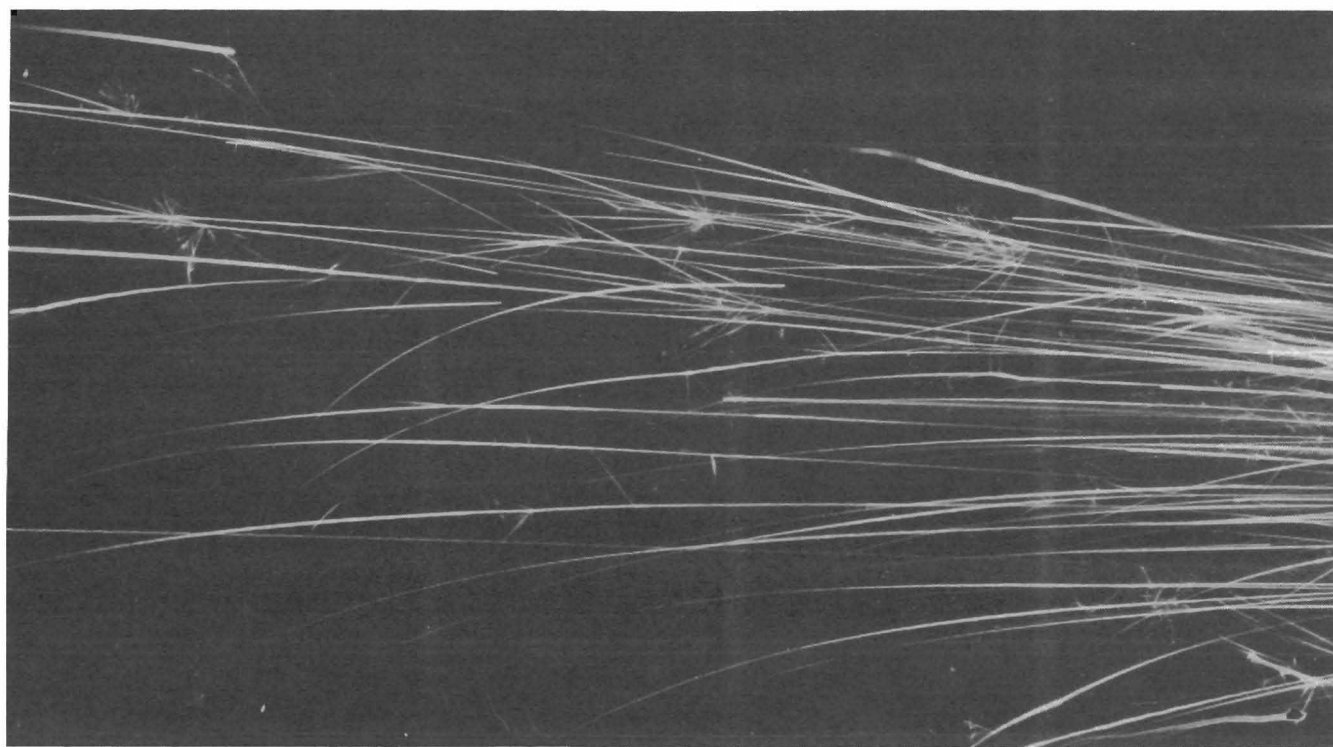
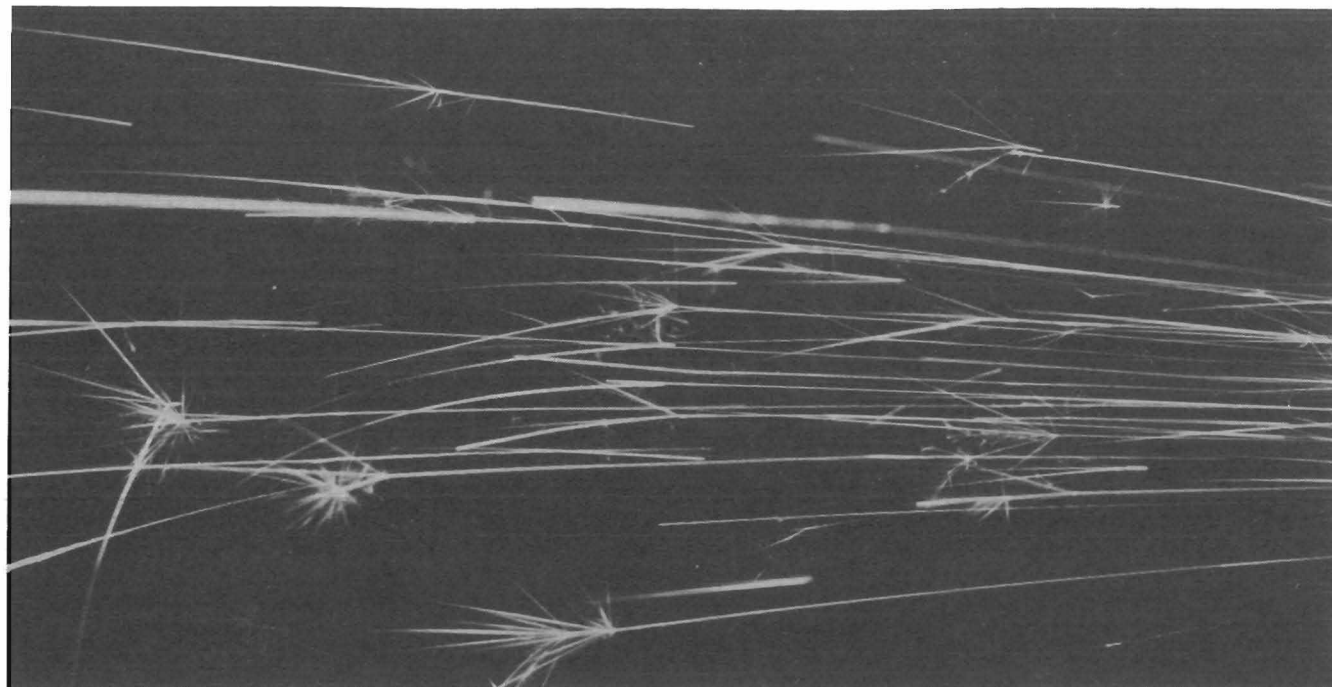


FIGURE 11. - Photographs of spark patterns showing the effect of carbide formers. *A*, 1020 carbon steel; *B*, 201 stainless steel.

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